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Continuous Beneficiation of Dolomitic Phosphate Ores

By B. E. Davis, T. O. Llewellyn,
and C. W. Smith



UNITED STATES DEPARTMENT OF THE INTERIOR

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UNITED STATES DEPARTMENT OF THE INTERIOR
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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

ft	foot	lb	pound
ft ³	cubic foot	lb/h	pound per hour
ft/s	foot per second	lb/ton	pound per ton
gal	gallon	min	minute
h	hour	pct	percent
in	inch	wt pct	weight percent
L	liter		

CONTINUOUS BENEFICIATION OF DOLOMITIC PHOSPHATE ORES

By B. E. Davis,¹ T. O. Llewellyn,² and C. W. Smith³

ABSTRACT

Over 80 pct of domestic phosphate rock, which is the starting material for phosphorus-containing fertilizers, comes from central Florida deposits. As higher grade deposits are depleted, lower grade phosphate ores containing MgO in the form of dolomite are encountered. Treating high-MgO ores with present beneficiation technology will not yield a concentrate meeting specifications for acidulation. The Bureau of Mines conducted research on three high-MgO phosphate samples from south Florida. The samples contained, in percent, 5.4 to 10.2 P_2O_5 , 1.9 to 7.6 MgO, and 35.3 to 60.1 acid insolubles. A technique of sizing, grinding, desliming, scrubbing, and redesliming was used to produce a feed suitable for continuous flotation studies. A conventional fatty acid-fuel oil rougher flotation followed by two to three cleaner stages resulted in concentrates containing, in percent, 30.7 to 31.3 P_2O_5 , 0.6 to 1.3 MgO, and 2.5 to 4.8 acid insolubles. The P_2O_5 recovery from the flotation feed ranged from 55.1 to 89.7 pct.

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INTRODUCTION

A basic requirement for survival of all living creatures is food. The future of our country could rest upon the ability of our farmers to efficiently grow crops to feed our citizens. For maximum crop yield, farmland must be fertilized. Phosphate rock is the raw material from which phosphorus-containing fertilizers are produced. Phosphate rock is also smelted in electric furnaces where elemental phosphorus is produced. Elemental phosphorus is reoxidized to produce high-grade phosphoric acid or converted into anhydrous derivatives. The furnace-grade acid is used for food-grade dicalcium phosphate and sodium tripolyphosphate in detergents (1).⁴ Of the 53.6 million metric tons of phosphate rock produced in the United States in 1981, 86.3 pct was produced in Florida and North Carolina (1). The importance of Florida phosphate is self-evident.

Currently phosphate is mined from the Bone Valley Formation in central Florida. Overburden is stripped with a dragline until the phosphate ore, known as matrix, is reached. The matrix is mined with the dragline and dumped into a slurry pit. The matrix is slurried with high-pressure hydraulic jets and pumped via a pipeline to the beneficiation plant several miles away. At the beneficiation plant, the matrix is washed and sized at 14 mesh. The plus 14-mesh material, called pebble, is a salable product and needs no further beneficiation. The minus 14-mesh slurry is deslimed at 150 mesh in hydrocyclones, producing primary slimes. The 14- by 150-mesh material is sized into two fractions, generally 14 by 28 mesh and 28 by 150 mesh, for anionic flotation of the phosphate. The two phosphate float products are combined and de-oiled to remove the fatty acid-fuel oil collector. The dereagentized material is then conditioned with a cationic collector; the remaining silica is removed as the float product, and the phosphate concentrate is

removed as the underflow. This process is known as the double-float method and was patented by Arthur Crago in 1940 (2).

U.S. resources of phosphate rock, which amount to billions of tons, are ample to supply domestic needs for centuries (1, 3). Mine and plant capacities are adequate to meet present demand, and expansion of the industry can be expected to meet future demand. However, as higher grades deposits are depleted and the phosphate mining industry moves into south Florida, it encounters lower grade ore bodies. The amount of coarse particles suitable for phosphate pebble is also drastically reduced. The P_2O_5 content is lower, and the amount of MgO increases owing to the presence of carbonate minerals (4). At present high-MgO ores are bypassed during mining operations because today's beneficiation technology will not yield a concentrate suitable for acidulation. When phosphate concentrates are acidulated to produce phosphoric acid, superphosphate, and triple superphosphate, the carbonate minerals consume sulfuric acid, resulting in a lower P_2O_5 production rate with a higher consumption of sulfuric acid and defoamer (5-6). High amounts of MgO make gypsum filtration difficult. An insoluble magnesium salt precipitates in the phosphoric acid; it settles during shipment and storage, creating handling problems. Its presence also lowers the P_2O_5 content of the finished fertilizer products. The phosphate industry has not set definite maximum MgO limits. However, it is generally accepted that most acidulation plants can handle up to 1.0 pct MgO.

The main mineral constituents in these ores are quartz, collophane, and dolomite. The two latter minerals respond similarly to known flotation processes so that separation is difficult. Several processes have been described in the literature for separation of phosphate and dolomite. One such process involved reagentizing a concentrate from the double-float method with hydrofluoric acid and an amine collector at pH 5.4. The

⁴Underlined numbers in parentheses refer to items in the list of references at the end of this report.

reagentized concentrate was subjected to rougher, cleaner, recleaner, and recleaner phosphate flotations (7). Another process described in the literature consisted of reagentizing a concentrate from the double-float method with a carbonate collector, a phosphate depressant, and a pH regulator. The dolomite was removed as the overflow product, and the phosphate concentrate was the underflow (8).

Laboratory beneficiation studies have been completed and reported for two south

Florida high-MgO ores (9). The Bureau of Mines devised a technique of sizing, grinding, desliming, scrubbing, redesliming, and flotation to treat high-MgO ores. The flotation involved using a conventional fatty acid-fuel oil phosphate rougher flotation and two or three cleaner flotations to produce a concentrate. To determine the feasibility of this process, the Bureau has conducted continuous beneficiation studies on three south Florida high-MgO phosphate ores. This report summarizes the results of these studies.

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Chemical Co., Mulberry, FL; John W. Kraus, W. R. Grace and Co., Bartow, FL; and Ed Finch, formerly of Amax Chemical Corp., Lakeland, FL.

SAMPLE DESCRIPTION

Three samples of high-MgO phosphate matrix from south Florida were provided by three different companies that have land leases in high-MgO areas. The matrix was shipped to the Bureau's Tuscaloosa Research Center in open dump trucks and contained about 15 pct moisture, which is about that of the original bed. Approximately 10 to 16 tons of sample were provided by each company. The matrix was protected and kept moist for processing. Representative samples were taken from each matrix for analysis and laboratory studies. The matrix samples were mostly fine to medium-sized sand but contained some large rocks and aggregates up to 1 ft in diameter. Samples of the large rocks were analyzed and found to be mostly dolomite. Chemical analysis of the matrix samples showed that they contained, in percent, 5.4 to 10.2 P_2O_5 , 1.9 to 7.6 MgO, and 35.3 to 60.1 insolubles. Table 1 gives the complete chemical analysis of the samples.

TABLE 1. - Chemical analysis of phosphate samples, percent

	Sample 1	Sample 2	Sample 3
P_2O_5	10.2	5.4	6.6
CaO.....	15.5	15.4	24.4
MgO.....	1.9	3.5	7.6
CO ₂	4.8	11.6	18.6
Insol.....	58.2	60.1	35.3
Al ₂ O ₃8	1.7	2.6
Fe ₂ O ₃	1.1	1.9	1.8

Samples of the matrix were crushed to pass 3 mesh, and size analysis was performed. All three samples were similar in that most of the dolomite reported to the coarse and fine fractions, as can be seen by the MgO analysis and distribution given in tables 2-4. By desliming at 150 mesh, 40.2 pct of the MgO in sample 1, 40.4 pct of the MgO in sample 2, and 65.6 pct of the MgO in sample 3 can be removed. The accompanying loss of P_2O_5 would be 7.0 pct, 10.0 pct, and 8.2 pct, respectively.

TABLE 4. -- Particle size and chemical analysis of sample 3, percent

Size fraction, mesh	Wt pct	P ₂ O ₅	CaO	MgO	CO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
ANALYSIS								
Plus 14.....	25.4	14.4	37.9	6.4	15.5	17.5	1.8	1.8
Minus 14 plus 20.....	1.4	17.2	37.6	4.2	13.9	19.8	1.6	2.0
Minus 20 plus 28.....	1.7	14.9	33.0	4.3	11.5	25.9	1.7	1.8
Minus 28 plus 35.....	4.5	10.1	19.0	1.3	4.7	56.2	1.2	1.4
Minus 35 plus 48.....	12.5	7.2	12.9	.9	3.2	68.2	1.1	.9
Minus 48 plus 65.....	12.8	8.8	15.8	.9	3.6	62.7	1.1	1.0
Minus 65 plus 100.....	6.1	11.2	20.7	1.8	5.4	52.0	1.4	1.2
Minus 100 plus 150.....	2.2	4.8	14.7	4.4	8.6	54.7	2.0	1.5
Minus 150 plus 200.....	2.2	1.6	28.1	14.9	18.7	18.3	2.5	2.3
Minus 200 plus 400.....	9.1	1.3	38.2	16.9	22.3	7.4	2.3	2.4
Minus 400.....	22.1	2.3	18.2	11.1	22.1	24.9	7.9	2.3
Composite.....	100.0	8.1	24.9	6.6	13.2	35.2	3.0	1.7
DISTRIBUTION								
Plus 14.....	25.4	45.3	38.6	24.8	29.7	12.6	15.3	26.9
Minus 14 plus 20.....	1.4	3.0	2.1	.9	1.5	.8	.8	1.6
Minus 20 plus 28.....	1.7	3.1	2.3	1.1	1.5	1.3	1.0	1.8
Minus 28 plus 35.....	4.5	5.6	3.4	.9	1.6	7.2	1.8	3.7
Minus 35 plus 48.....	12.5	11.1	6.5	1.7	3.0	24.3	4.6	6.6
Minus 48 plus 65.....	12.8	13.9	8.1	1.8	3.5	22.8	4.7	7.5
Minus 65 plus 100.....	6.1	8.5	5.1	1.7	2.5	9.0	2.9	4.3
Minus 100 plus 150.....	2.2	1.3	1.3	1.5	1.4	3.4	1.5	1.9
Minus 150 plus 200.....	2.2	.4	2.5	5.0	3.1	1.1	1.9	3.0
Minus 200 plus 400.....	9.1	1.5	14.0	23.4	15.3	1.9	7.0	12.8
Minus 400.....	22.1	6.3	16.1	37.2	36.9	15.6	58.5	29.9
Total.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

Chemical analysis of the large rocks contained in the samples showed that they contained 9.4 to 19.1 pct MgO and only 1.7 to 6.1 pct P₂O₅. Based on these findings, the plus 1/2-in material was removed from samples 1 and 3 and the plus 1-in material was removed from sample 2, in batch and continuous processing studies. Table 5 gives the analysis of the oversize material.

Mineralogical examination of the samples showed that they consisted mostly of collophane, dolomite, and quartz. The subsieve material in the samples contained dolomite and the clay minerals

attapulgite, kaolinite, and montmorillonite. The minerals were considered liberated at 14 to 28 mesh.

TABLE 5. -- Chemical analysis of oversize waste, percent

	Sample 1	Sample 2	Sample 3
P ₂ O ₅	1.7	6.1	1.9
CaO.....	28.4	31.3	31.9
MgO.....	19.1	9.4	14.0
CO ₂	40.2	20.3	28.8
Insol.....	7.4	25.7	5.4
Al ₂ O ₃3	1.9	2.0
Fe ₂ O ₃	2.9	1.2	1.8

LABORATORY BATCH STUDIES

Batch flotation tests employing the Bureau technique previously reported were conducted on samples of the matrix (9). A flotation feed was prepared in the following manner: A weighed sample was screened at 14 mesh (samples 1 and 3) or 28 mesh (sample 2), and the oversize material was ground in a ball mill containing ceramic balls as the grinding medium. The ground material was recombined with the undersize material, and the slurry was deslimed at 150 mesh, producing the primary slimes. The deslimed pulp was scrubbed for 10 min using 1.0 lb of sodium hydroxide per ton of flotation feed.⁵ The scrubbed material was deslimed at 150 mesh, producing the scrubber slimes.

SAMPLE 1 RESULTS

The prepared flotation feed was conditioned at 40 pct solids with 1.6 lb/ton fatty acid-fuel oil (2:3 ratio) for 2 min at pH 9.2. A rougher phosphate concentrate was floated for approximately 2 min. The rougher concentrate was repulped and cleaned three times with 1.0 lb/ton sodium silicate added to each cleaner stage. Table 6 shows the operating conditions of a batch test for sample 1. The final concentrate contained, in percent, 30.7 P₂O₅, 0.5 MgO, and 4.4 SiO₂. P₂O₅ recovery from the flotation feed was 87.1 pct. Detailed results are presented in table 7.

⁵All reagent dosages are expressed in pound per ton of flotation feed.

SAMPLE 2 RESULTS

Prepared flotation feed from sample 2 was conditioned at 40 pct solids with 1.6 lb/ton fatty acid-fuel oil (2:3 ratio) for 2 min at pH 9.2. A rougher phosphate concentrate was floated for approximately 2 min. The rougher concentrate was cleaned three times using 1.0 lb/ton sodium silicate in each cleaner stage. Table 8 gives the operating conditions for a batch flotation test. The final concentrate contained, in percent, 27.5 P₂O₅, 2.0 MgO, and 4.2 SiO₂. The attendant P₂O₅ recovery from the flotation feed was 85.3 pct. Table 9 presents detailed results of a batch flotation test.

SAMPLE 3 RESULTS

Flotation feed prepared from sample 3 was conditioned at 40 pct solids for 2 min at pH 9.0 with 1.6 lb/ton fatty acid-fuel oil (2:3 ratio). A rougher phosphate concentrate was floated for approximately 2 min. The rougher concentrate was cleaned three times with 1.0 lb/ton sodium silicate added to each cleaner. Table 10 gives the operating conditions for batch flotation testing. The final concentrate contained, in percent, 29.3 P₂O₅, 1.1 MgO, and 6.9 SiO₂. The P₂O₅ recovery from the flotation feed was 89.1 pct. Complete results are shown in table 11.

TABLE 6. - Batch flotation conditions for sample 1

Operation	Condition		Reagent, lb/ton of flotation feed
	Time, min	pH	
Scrub.....	10	9.5	1.0 sodium hydroxide.
Condition.....	2	9.2	1.6 fatty acid-fuel oil.
Rougher flotation....	2	9.2	None.
Cleaner flotation 1...	1	9.2	1.0 sodium silicate.
Cleaner flotation 2...	1	9.2	1.0 sodium silicate.
Cleaner flotation 3...	1	9.2	1.0 sodium silicate.

TABLE 7. Batch flotation results for sample 1, percent

Product	Wt pct	P ₂ O ₅	CaO	MgO	CO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
ANALYSIS								
Concentrate.....	25.2	30.7	48.2	0.5	9.7	4.4	1.1	2.3
Cleaner tailings.....	10.6	8.4	13.7	.6	4.7	64.4	1.3	1.2
Rougher tailings.....	41.8	.6	.7	.1	1.7	92.1	1.0	.6
Minus 150-mesh scrubber slimes.....	2.7	8.4	15.8	1.8	11.6	39.7	8.0	3.3
Minus 150-mesh primary slimes	19.7	5.1	10.4	1.8	8.3	50.5	7.7	2.9
Composite.....	100.0	10.1	16.3	.6	5.6	57.5	2.6	1.6
DISTRIBUTION								
Concentrate.....	25.2	76.6	74.3	18.9	43.6	1.9	10.8	35.8
Cleaner tailings.....	10.6	8.8	8.8	10.1	8.9	11.8	5.4	7.8
Rougher tailings.....	41.8	2.5	1.8	6.7	12.7	67.1	16.3	15.6
Minus 150-mesh scrubber slimes.....	2.7	2.2	2.6	7.8	5.6	1.9	8.4	5.5
Minus 150-mesh primary slimes	19.7	9.9	12.5	56.5	29.2	17.3	59.1	35.3
Total.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

NOTE.--Recovery of P₂O₅ from flotation feed was 87.1 pct.

TABLE 8. - Batch flotation conditions for sample 2

Operation	Condition		Reagent, lb/ton of flotation feed
	Time, min	pH	
Scrub.....	10	9.5	1.0 sodium hydroxide.
Condition.....	2	9.2	1.6 fatty acid-fuel oil.
Rougher flotation.....	2	9.2	None.
Cleaner flotation 1....	1	9.2	1.0 sodium silicate.
Cleaner flotation 2....	1	9.2	1.0 sodium silicate.
Cleaner flotation 3....	1	9.2	1.0 sodium silicate.

TABLE 9. - Batch flotation results for sample 2, percent

Product	Wt pct	P ₂ O ₅	CaO	MgO	CO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
ANALYSIS								
Concentrate.....	13.0	27.5	49.1	2.0	9.8	4.2	1.1	2.0
Cleaner tailings.....	43.5	.6	2.0	.9	1.7	91.7	1.0	.4
Rougher tailings.....	16.9	2.1	5.2	.6	2.2	88.5	.9	.6
Minus 150-mesh scrubber slimes.....	2.2	9.9	24.1	3.1	7.6	42.2	1.5	1.8
Minus 150-mesh primary slimes	24.4	4.2	17.2	7.8	26.2	29.2	5.6	.9
Composite.....	100.0	5.4	12.9	2.7	8.9	63.4	2.1	.8
DISTRIBUTION								
Concentrate.....	13.0	65.8	49.7	9.5	14.2	0.9	6.7	32.7
Cleaner tailings.....	43.5	4.8	6.8	14.4	8.3	62.8	20.4	21.9
Rougher tailings.....	16.9	6.5	6.8	3.7	4.2	23.6	7.1	12.8
Minus 150-mesh scrubber slimes.....	2.2	4.0	4.1	2.5	1.9	1.5	1.5	5.0
Minus 150-mesh primary slimes	24.4	18.9	32.6	69.9	71.4	11.2	64.3	27.6
Total.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

NOTE.--Recovery of P₂O₅ from flotation feed was 85.3 pct.

TABLE 10. -- Batch flotation conditions for sample 3

Operation	Condition		Reagent, lb/ton of flotation feed
	Time, min	pH	
Scrub.....	10	9.0	1.0 sodium hydroxide.
Condition.....	2	9.0	1.6 fatty acid-fuel oil.
Rougher flotation.....	2	9.0	None.
Cleaner flotation 1....	1	9.0	1.0 sodium silicate.
Cleaner flotation 2....	1	9.0	1.0 sodium silicate.
Cleaner flotation 3....	1	9.0	1.0 sodium silicate.

TABLE 11. - Batch flotation results for sample 3, percent

Product	Wt pct	P ₂ O ₅	CaO	MgO	CO ₂	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃
ANALYSIS								
Concentrate.....	17.4	29.3	47.2	1.1	8.5	6.9	1.1	1.6
Cleaner tailings.....	15.8	2.4	5.7	1.3	4.2	80.0	1.2	.4
Rougher tailings.....	19.8	1.2	2.9	.5	3.4	86.1	1.2	.3
Minus 150-mesh scrubber slimes.....	5.8	4.9	31.0	13.0	30.7	11.7	3.5	2.6
Minus 150-mesh primary slimes	41.2	2.6	25.1	12.9	24.3	17.4	4.9	3.2
Composite.....	100.0	7.1	21.8	6.6	14.6	38.7	2.8	1.9
DISTRIBUTION								
Concentrate.....	17.4	72.0	37.6	2.9	10.1	3.1	6.7	14.9
Cleaner tailings.....	15.8	5.4	4.1	3.1	4.5	32.6	6.7	3.4
Rougher tailings.....	19.8	3.4	2.6	1.5	4.6	44.0	8.4	3.2
Minus 150-mesh scrubber slimes.....	5.8	4.0	8.2	11.5	12.2	1.8	7.1	8.1
Minus 150-mesh primary slimes	41.2	15.2	47.5	81.0	68.6	18.5	71.1	70.4
Total.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

NOTE.--Recovery of P₂O₅ from scrubbed flotation feed was 89.1 pct.

CONTINUOUS PROCESSING STUDIES

FLOTATION FEED PREPARATION

Upon completion of characterization and batch beneficiation studies, a continuous processing plant was designed and assembled. A 375-gal tank was used to slurry the matrix. The tank was equipped with two triple-bladed 12-in-diam impellers mounted on a single shaft for mixing. A 1/2-in grizzly was placed on top of the tank for removal of oversize (1-in grizzly for sample 2). The matrix was dumped on the grizzly and washed with a high-pressure firehose, and the oversize was removed. The slurry was mixed in the tank and pumped to a vibrating screen fitted with a 14-mesh screen (28 mesh for sample 2). The screen oversize was fed

to a ball mill with a peripheral discharge and containing ceramic balls as the grinding medium. The mill was operated in closed circuit with the vibrating screen so that all the material passed the screen size. The slurry was pumped from the screen discharge to a spiral classifier for removal of the minus 150-mesh primary slimes. The deslimed pulp was fed to a scrubber to break up soft dolomite and clay aggregates and to clean the mineral surfaces. The scrubber was a square cross-sectional type with two four-bladed impellers with a blade tip speed of approximately 7.8 ft/s. Pulp density in the scrubber was 50 pct solids. Retention time required in the scrubber was approximately 5 min, about

half that required in batch tests. Sodium hydroxide was added to the scrubber for dispersion in the amounts of 0.8 lb/ton for sample 1, 1.8 lb/ton for sample 2, and 0.9 lb/ton for sample 3. The scrubber discharge was pumped to a spiral classifier, and the minus 150-mesh scrubber slimes were removed. Figure 1 shows the flow diagram for the flotation feed preparation.

Tables 12-14 summarize the results of the flotation feed preparation. By removing the oversize, desliming at 150 mesh, scrubbing, and redesliming, 91.2 pct of the MgO was rejected from sample 1, 83.1 pct from sample 2, and 95.4 pct from sample 3. The resultant loss of P_2O_5 was 19.6 pct for sample 1, 28.5 pct for sample 2, and 37.4 pct for sample 3. Sample 3 contained an unusually large amount (27.5 pct) of P_2O_5 in the primary slimes.

CONTINUOUS FLOTATION CIRCUIT

The discharge from the spiral classifier used for removing the scrubber slimes was fed directly to a conditioner for addition of reagents. The flotation feed was conditioned with sodium carbonate for pH control, fatty acid-fuel oil as a phosphate collector, and frother for a manageable froth. Pulp residence time was approximately 2 min. The fatty

acid and fuel oil were mixed in a 2:3 ratio. Dowfroth 250⁶ was the frother used. All three reagents were added simultaneously. Pulp density in the conditioner was 35 to 40 pct solids. A pH of 9.2 was maintained in the flotation circuit.

Flotation was accomplished in a bank of six flotation cells with an effective cell volume of 7 L each. Two cells were used for rougher flotation and two cells for the first cleaner stage. One cell each was used for the remaining cleaner stages. After several continuous tests it was discovered that only two cleaner stages were needed for samples 1 and 3, as opposed to three cleaners in batch tests. Sample 2 required three cleaners. Sodium silicate was added to each cleaner stage for gangue depression. For samples 1 and 3 the tailings from the recleaner were recirculated to the cleaner stage. For sample 2, which required three cleaners, the re-recleaner tailings were recirculated to the recleaner stage and the recleaner tailings to the cleaner stage. Figure 2 is a flow diagram of the flotation circuit. Figure 3 shows the flotation cells in operation.

⁶Reference to specific products does not imply endorsement by the Bureau of Mines.

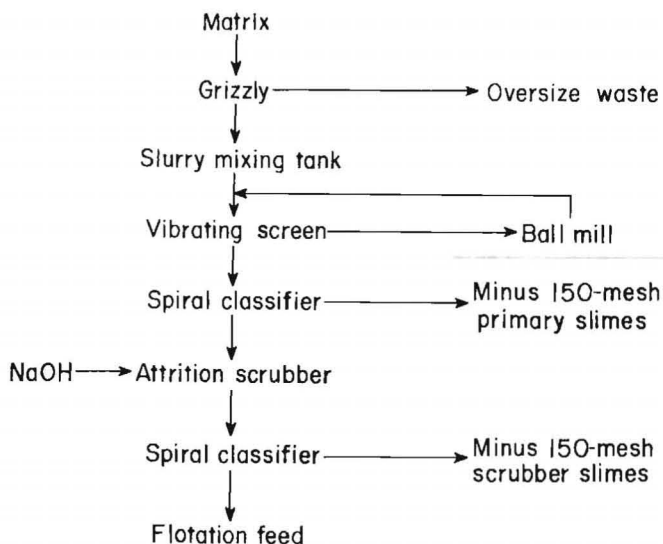


FIGURE 1. - Flow diagram of continuous flotation feed preparation.

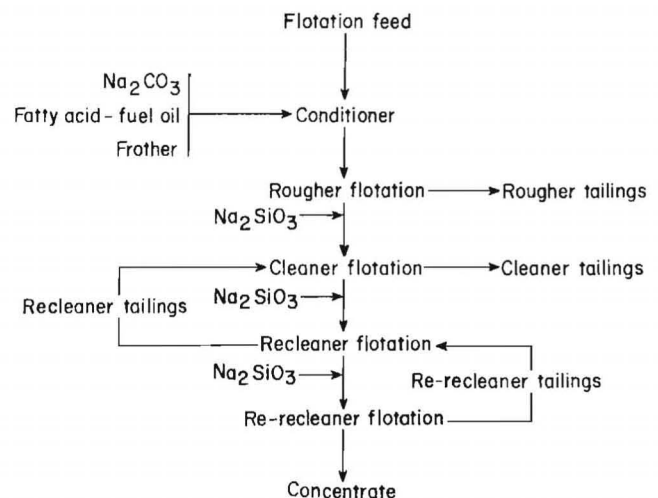


FIGURE 2. - Flow diagram of continuous flotation circuit.

TABLE 12. - Flotation feed preparation results for sample 1, percent

[illegible]

TABLE 13. - Flotation-feed preparation results for sample 2, percent

[illegible]

TABLE 14. - Flotation feed preparation results for sample 3, percent

[illegible]

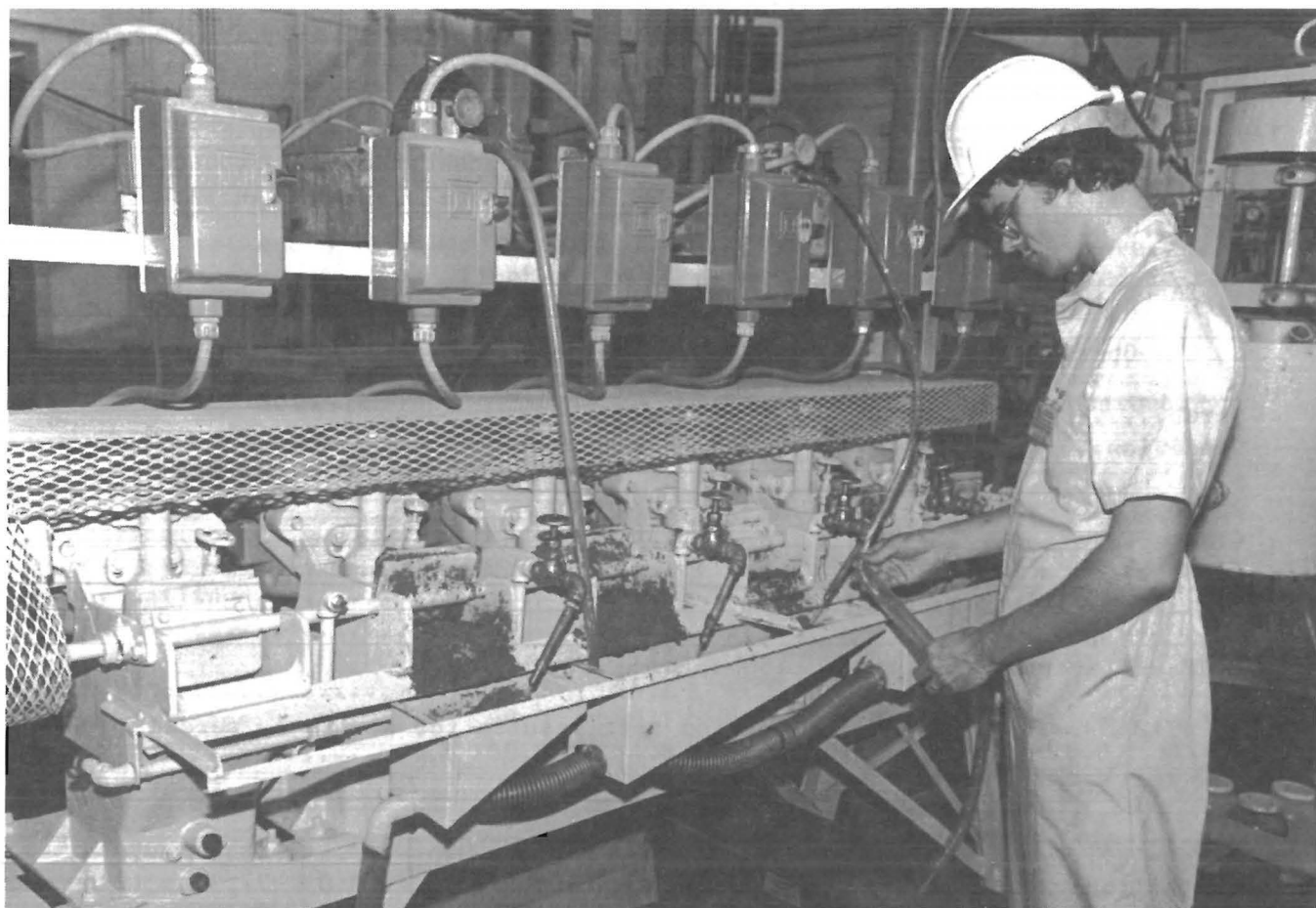


FIGURE 3. - Continuous flotation cells in operation.

SAMPLE 1 RESULTS

Continuous flotation tests using sample 1 were conducted with a flotation feed rate of approximately 150 lb/h. After the flotation circuit reached steady state, the products were sampled and collected for up to 4 h. In a typical test, flotation feed was conditioned with 0.9 lb/ton sodium carbonate, 2.8 lb/ton fatty acid-fuel oil, and 0.04 lb/ton frother. A rougher phosphate concentrate was floated and cleaned two times with 0.8 lb/ton sodium silicate added to each cleaner. Table 15 presents detailed operating conditions. Rougher flotation time was approximately 5 min at 30 pct solids. Table 16 gives the results of the continuous test. The final

concentrate contained, in percent, 31.3 P_2O_5 , 47.7 CaO, 0.6 MgO, 7.5 CO_2 , 3.5 insolubles, 1.1 Al_2O_3 , and 2.1 Fe_2O_3 . The attendant P_2O_5 recovery was 89.7 pct. These results were comparable to batch flotation results.

SAMPLE 2 RESULTS

Continuous flotation tests using sample 2 required a longer flotation time than for samples 1 and 3. As a result, the flotation feed rate was decreased to 85 lb/h, for a rougher flotation time of approximately 8 min at 30 pct solids. The flotation reagents were added to the conditioner in the amounts of 1.5 lb/ton sodium carbonate, 3.0 lb/ton fatty acid-fuel oil, and 0.08 lb/ton frother. A

phosphate rougher concentrate was floated and cleaned three times. Sodium silicate was added to each cleaner stage in the amount of 1.5 lb/ton. Table 17 gives detailed operating conditions. The final concentrate contained, in percent, 30.7 P₂O₅, 49.6 CaO, 1.3 MgO, 8.6 CO₂, 2.5 insolubles, 1.0 Al₂O₃, and 2.1 Fe₂O₃. The

attendant P₂O₅ recovery was 55.1 pct. Recovery was improved in other tests but only with lower P₂O₅ and higher MgO content in the concentrate. Table 18 presents results for sample 2. A better grade concentrate was produced in continuous processing than in batch tests.

TABLE 15. - Operating conditions for continuous flotation of sample 1

Operation	Condition		Reagent, lb/ton of flotation feed
	Time, min	pH	
Scrub.....	5	9.5	0.8 sodium hydroxide.
Condition.....	2	9.2	0.9 sodium carbonate, 2.8 fatty acid-fuel oil, 0.04 frother.
Rougher flotation.....	Nap	9.2	None.
Cleaner flotation 1....	Nap	9.2	0.8 sodium silicate.
Cleaner flotation 2....	Nap	9.2	0.8 sodium silicate.
Nap Not applicable.			

NOTE.--Flotation feed rate: 150 lb/h.

TABLE 16. - Continuous flotation test results for sample 1, percent

Product	Wt pct	P ₂ O ₅	CaO	MgO	CO ₂	Insol	Al ₂ O ₃	Fe ₂ O ₃
ANALYSIS								
Concentrate.....	31.2	31.3	47.7	0.6	7.5	3.5	1.1	2.1
Cleaner tailings.....	13.8	6.6	10.6	.2	3.0	77.3	1.2	1.0
Rougher tailings.....	55.0	.4	.6	.1	.7	98.1	1.1	.5
Composite.....	100.0	10.9	16.7	.3	3.1	65.7	1.1	1.1
DISTRIBUTION								
Concentrate.....	31.2	89.7	89.3	77.6	74.6	1.7	30.9	61.4
Cleaner tailings.....	13.8	8.3	8.7	14.9	13.1	16.2	14.8	12.9
Rougher tailings.....	55.0	2.0	2.0	7.5	12.3	82.1	54.3	25.7
Total.....	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

TABLE 17. - Operating conditions for continuous flotation of sample 2

Operation	Condition		Reagent, lb/ton of flotation feed
	Time, min	pH	
Scrub.....	5	9.5	1.8 sodium hydroxide.
Condition.....	2	9.2	1.5 sodium carbonate, 3.0 fatty acid-fuel oil, 0.08 frother.
Rougher flotation.....	Nap	9.2	None.
Cleaner flotation 1....	Nap	9.2	1.5 sodium silicate.
Cleaner flotation 2....	Nap	9.2	1.5 sodium silicate.
Cleaner flotation 3....	Nap	9.2	1.5 sodium silicate.
Nap Not applicable.			

NOTE.--Flotation feed rate: 85 lb/h.

CONCLUSIONS

A method to treat high-MgO south Florida phosphate ores was devised by the Bureau of Mines. The method employed sizing, grinding, desliming, scrubbing, redesliming, and flotation to produce a phosphate concentrate. A continuous processing plant was designed and assembled to test the method on a continuous basis. Three different samples were obtained and processed. The samples contained, in

percent, 5.4 to 10.2 P_2O_5 , 1.9 to 7.6 MgO, and 35.3 to 60.1 insolubles. Concentrates were produced from these samples that contained, in percent, 30.7 to 31.3 P_2O_5 , 0.6 to 1.3 MgO, and 2.5 to 4.8 insolubles. The P_2O_5 recovery from the flotation feed ranged from 55.1 to 89.7 pct. It is concluded that the Bureau method is a feasible means for processing high-MgO ores.

REFERENCES

1. Stowasser, W. F. Phosphate. Bu-Mines Mineral Commodity Profile, 1983, 18 pp.
2. Crago, A. (assigned to Phosphate Recovery Corp., New York, NY). Process of Concentrating Phosphate Minerals. U.S. Pat. 2,293,640, Oct. 8, 1940.
3. Catchcart, J. B., and R. A. Gulbrandsen. Phosphate Deposits. U.S. Geol. Surv. Prof. Paper 820, 1973, pp. 515-525.
4. Lawver, J. E., B. L. Murowchick, and R. E. Snow. Beneficiation of South Florida High Carbonate Phosphorites. Pres. at ISMA 1978 Technical/Economic Conf., Orlando, FL, Oct. 23-27, 1978. Fertilizer Technology Preprint TA/78/1, pp. 1-15.
5. Bushell, C. H. G., and H. E. Hirsch (assigned to Cominco Ltd., Montreal, Quebec, Canada). Flotation Process. U.S. Pat. 3,462,017, Aug. 19, 1969.
6. Kouloheris, A. P. Solving Problems in Chemical Processing of Low Quality Phosphate Rock. Eng. and Min. J., v. 178, No. 9, 1977, pp. 104-108.
7. Snow, R. E. (assigned to International Minerals and Chemical Corp., Terre Haute, IN). Beneficiation of Phosphate Ore. U.S. Pat. 4,144,969, Mar. 20, 1979.
8. Lawver, J. E., W. O. McClintock, and R. E. Snow (assigned to International Minerals and Chemical Corp., Terre Haute, IN). Method of Beneficiating Phosphate Ores Containing Dolomite. U.S. Pat. 4,372,843, Feb. 8, 1983.
9. Llewellyn, T. O., B. E. Davis, G. V. Sullivan, and J. P. Hansen. Beneficiation of High-Magnesium Phosphate From Southern Florida. BuMines RI 8609, 1982, 16 pp.